

Claims:

An integrated process for preparation of sulphate of potash (SOP) from bittern comprising:

- 5 (i) subjecting bittern to fractional crystallization to obtain kainite type mixed salt with high kainite content and $MgCl_2$ -rich end bittern, and subjecting the $MgCl_2$ rich end bittern to desulphation;
 - (ii) treating the kainite type mixed salt with water and mother liquor obtained in step (xiii) below to leach out substantially all $NaCl$ from the mixed salt and simultaneously convert kainite into schoenite;
 - 10 (iii) filtering the schoenite and separating the filtrate;
 - (iv) desulphating the filtrate with aqueous $CaCl_2$;
 - (v) filtering the gypsum produced in step (iv) and mixing the filtrate with the $MgCl_2$ -rich filtrate obtained in step (vii) below;
 - (vi) evaporating the resultant solution of step (v) and cooling to ambient temperature to crystallize crude carnallite;
 - 15 (vii) centrifuging the crude carnallite and recycling the required quantity of filtrate to step (v);
 - (viii) decomposing the crude carnallite with appropriate quantity of water from step (vi) to yield crude KCl and carnallite decomposed liquor;
 - 20 (ix) filtering the crude KCl , and washing with water to remove adhering $MgCl_2$ and subjecting to hot leaching for production of MOP and $NaCl$;
 - (x) mixing the carnallite decomposed liquor from step (viii) and washing from step (ix) and treating with hydrated lime;
 - (xi) filtering the slurry and washing the cake to produce $Mg(OH)_2$ and $CaCl_2$ -containing filtrate for the desulphation process of step (iv);
 - 25 (xii) treating by known method the schoenite produced in step (iii) with MOP produced in step (ix) to produce SOP under ambient condition;
 - (xiii) filtering the SOP and collecting separately the mother liquor hereinafter referred to as KEL;
 - 30 (xiv) recycling the KEL of step (xiii) in the process of step (ii).
2. A process as claimed in Claim 1 wherein the bittern contains K, Mg and SO_4 in concentrations that make it suitable for kainite production
 3. A process as claimed in claim 2 wherein said bittern is selected from sea bittern and sub-soil bittern, and preferably bittern with higher potassium content and which

also requires the least evaporation to produce kainite-type mixed salt, and waste bittern sources such as debrominated bittern effluent.

4. A process as claimed in any one of claims 1 to 3 wherein said mixed salt contains KCl-15-22%, NaCl-15-22%, MgSO_4 -28-40%, MgCl_2 -5-10%.
5. A process as claimed in any preceding claim wherein one part by weight of mixed salt is treated with 0.75-1.25 parts by volume of KEL and 0.3-0.7 parts by volume of water.
6. A process as claimed in any preceding claim wherein said KEL contains typically 15-17% KCl, 1-3% NaCl, 10-12% MgSO_4 , and 2-3% MgCl_2 .
7. A process as claimed in in any preceding claim wherein the SEL contains typically 8-10% KCl, 6-12% NaCl, 12-14% MgSO_4 and 5-7% MgCl_2 .
8. A process as claimed in any preceding claim wherein the schoenite contains typically 40-45% K_2SO_4 , 30-35% MgSO_4 and 0.5-2.0% NaCl.
9. A process as claimed in any preceding claim wherein the stoichiometric ratio of CaCl_2 to sulphate for the desulphatation reaction of step (iv) is 1.1:1 to 0.9:1, preferably 1:1.
10. A process as claimed in claim 7 wherein 1 part by volume of desulphated SEL was mixed with 0.5-1.5 parts by volume of MgCl_2 -rich end bittern of 36-38°Be' (s.g. 1.33-1.38), preferably, 0.7-0.9 parts of end bittern of 37 °Be' (sp. gr. 1.342), and more preferably, MgCl_2 -rich end bittern containing no sulphate.
11. A process as claimed in any one of claims 1 to 10 wherein the concentration of desulphated SEL to produce carnallite is carried out in a solar pan or in a multiple effect evaporator with simultaneous recovery of water.
12. A process as claimed in any preceding claim wherein evaporation is continued till the solution attained a temperature in the range of 120-128°C and more preferably, 122-124°C.
13. A process claimed in any preceding claim wherein the carnallite obtained has 15-20% KCl, 15-20% NaCl and 28-32% MgCl_2 .
14. A process claimed in any preceding claim wherein one part by weight of the carnallite is decomposed with 0.4-0.6 parts by volume of water, followed by washing of the cake with a small quantity of water.
15. A process claimed in any preceding claim wherein the molar ratio of active lime to MgCl_2 for production of $\text{Mg}(\text{OH})_2$ and CaCl_2 is in the range of 0.8-1.0, preferably, 0.90.

16. A process claimed in any preceding claim wherein said $\text{Mg}(\text{OH})_2$ obtained is calcined to produce MgO with 94-98 % purity and with 0.02-0.04 % B_2O_3 .
17. A process claimed in any preceding claim wherein the $\text{Mg}(\text{OH})_2$ is used without upgradation for neutralization of acidified de-brominated bittern where such bittern is used as potash source.
18. A process claimed in any preceding claim wherein the MOP obtained upon hot leaching of crude KCl has a purity in the range of 92-98% and NaCl content of 1-5 % , preferably >95% KCl and <2% NaCl .
19. A process as claimed in claims 1-3 wherein the NaCl obtained on hot leaching of crude KCl contains > 97% NaCl .
20. A process claimed in any preceding claim wherein one part by weight of schoenite is treated with 0.3-0.6 parts by weight of MOP and 1-2 parts by volume of water, and more preferably 0.4 parts by weight of MOP and 1.5 parts by volume of water, in the ambient temperature range of 20-45 degree C.
21. A process claimed in any preceding claim wherein the SOP produced has K_2O content in the range of 50-52% and chloride in the range of 0.5-2.0 %.